

PATENT ABSTRACTS OF JAPAN

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(21)Application number : 06-154787

(71)Applicant : MITSUBISHI CHEM CORP

(22)Date of filing : 06.07.1994

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(54) EPOXY RESIN COMPOSITION AND PREPREG PREPARED THEREFROM

(57)Abstract:

PURPOSE: To obtain the rein composition which can produce a prepreg that is excellent in storage atability at room temperature, is capable of being molded at relatively low temperatures, and causes no defects on the ground surface of its molded articles by incorporating a low-temperature-active potential curing agent and a specific whisker in an epoxy resin.

CONSTITUTION: This epoxy resin composition for prepreg is prepared by mixing 100 pts.wt. of an epoxy resin (A), 3-60 pts.wt. of a low-temperature-active potential curing agent (B) (e.g. an amine-adduct potential curing agent and a microcapsule potential curing agent of a diaphragm-breaking type, which are activated at about 80°C) and 0.05-30 pts.wt. of a whisker (C) with an aspect ratio of 3-500. The prepreg prepared from this resin composition has a proper workability and excellent composite characteristics, is excellent in storage stability at room temperature, is capable of being molded at a relatively low temperature around 80°C, produces no wastes upon molding, and causes no defects on the ground and coating surface of its molded articles.

LEGAL STATUS

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DERWENT-ACC-NO: 1996-124203

DERWENT-WEEK: 199613

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TITLE: Epoxy! resin compsn., useful for sports goods, e.g. fishing rods - contains low temp. active latent curative and whisker with specified aspect ratio, giving good keeping stability, impact resistance, etc.

PATENT-ASSIGNEE: MITSUBISHI CHEM CORP[MITU]

PRIORITY-DATA: 1994JP-0154787 (July 6, 1994)

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PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAN-IPC
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APPLICATION-DATA:

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JP 08020708A	N/A	1994JP-0154787	July 6, 1994

INT-CL (IPC): C08G059/40, C08J005/24, C08K007/04, C08L063/00

ABSTRACTED-PUB-NO: JP 08020708A

BASIC-ABSTRACT:

An epoxy resin compsn. contains (A) an epoxy resin and (B) 360 pts. wt. of a low temp. active latent curative and (C) 0.0530 pts. wt. of a whisker with aspect ratio of 3-500 per 100 pts. wt. (A).

Also claimed are prepregs using the epoxy resin compsns.

Pref. (B) is amine adduct type latent curative and microcapsulated latent curative. (C) comprises aluminium borate and calcium carbonate.

USE - The epoxy resin compsns. are useful for sports goods, esp. fishing rods and golf shafts, structural materials for industrial prods., cars, airplanes and ships and electronic civil engineering and building materials.

ADVANTAGE - The epoxy resin compsns. have excellent keeping stability and less resin flow on moulding without heat sag on tubular moulding, the prepregs can be moulded at a low temp. of 80deg.C, have excellent winding workability such as tack and drapability, thus improved productivity and impact resistance and can give large mouldings and the ground surface of a moulding obtd. using the prepregs has no defects such as flaws.

CHOSEN-DRAWING: Dwg.0/0

TITLE-TERMS: POLYEPOXIDE RESIN COMPOSITION USEFUL SPORTS GOODS FISH ROD CONTAIN LOW TEMPERATURE ACTIVE LATENT CURE WHISKER SPECIFIED ASPECT RATIO KEEP STABILISED IMPACT RESISTANCE

DERWENT-CLASS: A21

CPI-CODES: A05-A01B1; A08-D01; A08-R09; A12-S08;

ENHANCED-POLYMER-INDEXING:

Polymer Index [1.1]

018 ; G1570*R G1558 D01 D11 D10 D23 D22 D31 D42 D50 D69 D73 D83
F47 7A ; R00470 G1161 G1150 G1149 G1092 D01 D11 D10 D19 D18 D32
D50 D76 D93 F32 F30 ; P0464*R D01 D2 D42 F47 ; P1898*R P0464 D01

AN 1996:226040 HCAPLUS
 DN 124:291303
 ED Entered STN: 18 Apr 1996
 TI Epoxy resin compositions containing latent hardeners
 and whiskers, and prepregs with good tack and pliability from them
 IN Sato, Shoichi; Hino, Seiichi
 PA Mitsubishi Kagaku KK, Japan
 SO Jpn. Kokai Tokkyo Koho, 6 pp.
 CODEN: JKXXAF
 DT Patent
 LA Japanese
 IC ICM C08L063-00
 ICS C08G059-40; C08J005-24; C08K007-04
 CC 37-6 (Plastics Manufacture and Processing)
 FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI JP 08020708	A	19960123	JP 1994-154787	19940706
PRAI JP 1994-154787		19940706		

CLASS

PATENT NO.	CLASS	PATENT FAMILY CLASSIFICATION CODES
JP 08020708	ICM	C08L063-00
	ICS	C08G059-40; C08J005-24; C08K007-04
	IPCI	C08L0063-00 [ICM,6]; C08G0059-40 [ICS,6]; C08J0005-24 [ICS,6]; C08K0007-04 [ICS,6]
	IPCR	C08J0005-24 [I,C*]; C08J0005-24 [I,A]; C08G0059-00 [I,C*]; C08G0059-40 [I,A]; C08K0007-00 [I,C*]; C08K0007-04 [I,A]; C08L0063-00 [I,C*]; C08L0063-00 [I,A]

AB The compns. contain (A) epoxy resins 100, (B) latent hardeners active at low temperature 3-60, and (C) whiskers with aspect ratio 3-500 0.05-30 parts. The prepregs comprise reinforcing fibers impregnated with the resin compns. Thus, E 828 (bisphenol A epoxy resin) 70, E 1001 (bisphenol A epoxy resin) 30, PN 23 (amine adduct) 20, and Alborex YS 2 (Al borate whiskers) 5 parts were mixed and impregnated into Torayca T 300 to give prepregs showing resin flow 3.2%, 0° bending strength 175 kg/mm2, and 0° flexural modulus 13,700 kg/mm2.

ST epoxy resin hardener whisker prepreg

IT Crosslinking agents

Crystal whiskers

(epoxy resin compns. containing latent hardeners and whiskers for prepregs with good tack and pliability)

IT Carbon fibers, properties

RL: MOA (Modifier or additive use); PRP (Properties); USES (Uses)

(epoxy resin compns. containing latent hardeners and whiskers for prepregs with good tack and pliability)

IT Epoxy resins, properties

RL: POF (Polymer in formulation); PRP (Properties); USES (Uses)

(epoxy resin compns. containing latent hardeners and whiskers for prepregs with good tack and pliability)

IT Carbon fibers, properties

RL: MOA (Modifier or additive use); PRP (Properties); USES (Uses)

(graphite, Torayca T 300; epoxy resin compns.

containing latent hardeners and whiskers for prepregs with good tack and pliability)

IT 25068-38-6P 116723-05-8P 149676-34-6P

RL: IMF (Industrial manufacture); POF (Polymer in formulation); PRP (Properties); PREP (Preparation); USES (Uses)

(epoxy resin compns. containing latent hardeners and whiskers for prepregs with good tack and pliability)

IT 471-34-1, Whiscal, properties 11121-16-7, Alborex YS 3

RL: MOA (Modifier or additive use); PRP (Properties); USES (Uses)

aspect
ratio
spec. 3-500 0.05-30 parts

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention is excellent in handling workability, such as the moderate tuck nature (adhesiveness) of prepreg, and drape property (flexibility), and the preservation stability in a room temperature. Near 80 degree C can be comparatively fabricated at low temperature, and there are few resin flows at the time of shaping. It is related with the epoxy resin constituent suitable for the fiber consolidation composite which does not have furnace omission at the time of shaping of the tubular Plastic solid made of fiber consolidation resin, such as a fishing rod and a golf shaft, and does not have defects, such as a crack, in these Plastic solid polish front face especially and which has the outstanding composite-material property.

[0002]

[Description of the Prior Art] Fiber reinforced composite materials, such as a carbon fiber, are lightweight, have high intensity and a rate of high elasticity, are complex with resin, and the form of the so-called prepreg, for example, are widely used for industrial ingredients, such as the sport leisure fields, such as a shaft of a fishing rod and a golf club, and a tennis racket, a flat spring, and honeycomb structure material, and a pan at various applications, such as structural materials, such as automobile relation, an aircraft material, and a marine vessel, an electronic ingredient, and an engineering-works building material.

[0003] About shaping with which excelling in moderate handling workability, the preservation stability in a room temperature, and the mechanical property of a hardened material as a property required of such prepreg for fiber reinforced composite materials used shaping and pattern, and plastic pattern of large-scale structure material like [from the first in recent years] a marine vessel or a car, a hardening demand of a low-temperature short time is desired more, and the reduction demand of the shortening and energy cost which are a molding cycle is increasing.

[0004] By the way, the bulking agent which is 10 or less times [of the diameter of a reinforcement single fiber] particle size, for example, graphite powder, carbon black, an aluminium powder, silica powder, etc. are added to the things (JP,62-127317,A, JP,2-41314,A) using the epoxy resin as matrix resin of such [conventionally] prepreg for fiber reinforced composite materials, and these matrix resin, and that (JP,59-227931,A) which fell the fluidity of the matrix resin at the time of prepreg shaping is used for manufacture of prepreg.

[0005] Moreover, as prepreg for cold cure, it is proposed about the prepreg which used for JP,5-9262,A and JP,6-9802,A the latency curing agent 100 degrees C or less comparatively hardened at low temperature. In order that these matrix resin may not reduce preservation stability as compared with the conventional thing at the time of manufacture of prepreg, it is necessary to sink into consolidation fiber at low temperature more, therefore to set up resin viscosity lowness.

[0006] Especially the fiber reinforced composite material of the shape of tubing, such as a fishing rod and a golf shaft The mandrel with a taper which applied the release agent is received at a longitudinal direction in the prepreg which sank matrix resin into the sheet-like prepreg which lengthened and

arranged the carbon fiber and the glass fiber with the one direction, or the textiles of the above-mentioned consolidation fiber. Twist in the direction according to a desired design, and it twists spirally, using a tape winder etc. and making a heat shrink nature tape overlap little by little on it further. Subsequently, it puts into a heating furnace, matrix resin is hardened, giving the clamping force on a heat shrink nature tape, it takes out from a heating furnace and the tape after cooling is removed, and a deheart machine etc. draws out a mandrel and it is fabricating. However, in the conventional prepreg, the fault which prepreg moves toward a minor diameter side from the major-diameter side of a mandrel at the time of shaping that the so-called furnace omission occurred was produced. Since matrix resin hypoviscosity-izes and increases a fluidity with heating, if the bolting force on a heat shrink nature tape is added in such the condition, since the mandrel has the taper, component of a force arises from a major-diameter side in a minor diameter side, and the so-called furnace omission which prepreg moves in this direction generates this. If this furnace omission occurs, the unevenness of distribution will be made to matrix resin or consolidation fiber, and it will also become the cause of the defect of about [that desired composite-material physical properties are not acquired] and mold goods, or the curvature generating cause of a narrow diameter portion.

[0007] On the other hand, the yarn which sank yarn or thermosetting resin into JP,57-22636,A in advance of prepreg volume attachment by the mandrel is spirally twisted around the longitudinal direction of a mandrel, and the method of preventing furnace omission by the friction is proposed. However, about [that this approach is not enough as the furnace omission prevention effectiveness] and yarn remains in mold goods, and is not desirable on a property. Moreover, to the matrix resin for prepreps, before winding prepreg around a mandrel, when the gel time in 130 degrees C applies 80% or less of heat-curing resin of said resin to a mandrel, the method of preventing furnace omission is proposed by JP,59-159315,A. However, by this approach, the resin for undercoat which adjusted the gel time by B-stage-ization is required, in advance of shaping, the spreading process of this under coat resin and an air dried are required, and productivity is fallen remarkably. Furthermore, the curing temperature of 100 degrees C or more is required for such prepreps.

[0008] Moreover, about the handling nature at the time of the activity of prepreg (a tuck, drape property), since it becomes not only the productivity at the time of shaft winding but the cause of the defects on the front face of mold goods (crack etc.), a moderate property is required. If tuck nature is too strong, it will be easy to involve in Ayr at the time of winding, and if too low, workability, such as lamination of prepreg, will fall. Moreover, when drape property is imperfection, HANE is produced into the end part of a volume at the time of prepreg winding, and it also becomes the cause of a crease by the carbon fiber of the rate of high elasticity especially. However, these factors appear in a front face as defects, such as a crack, at the time of the polish after shaft shaping, and paint, and have become generating of a defective and the cause of lowering of physical properties, and the yield was reduced remarkably.

[0009] Thus, in the prepreg using the conventional matrix resin constituent, it was very difficult to offer the prepreg which the preservation stability in a room temperature is good, and hardens enough with shaping of 100 degrees C or less, does not have [has moderate workability and the outstanding composite-material property,] the furnace omission at the time of shaping, and does not have a defect in polish of mold goods, and a painted surface.

[0010]

[Problem(s) to be Solved by the Invention] The object of this invention solves the above-mentioned fault of the conventional approach, it can prevent the furnace omission at the time of shaping, without performing a head end process, and it can obtain the fiber reinforced composite material mold goods 100 degrees C or less which can fabricate at low temperature comparatively and have a desired property, without falling a composite-material property. Moreover, in the usual winding temperature, since the prepreg using the epoxy resin constituent of this invention has moderate winding workability and can control the resin flow at the time of shaping freely, it offers the outstanding Plastic solid which does not have defects (crack etc.) in the mold-goods front face after polish.

[0011]

[Means for Solving the Problem] That is, this invention consists in the prepreg using the epoxy resin constituent and this to which (B) low-temperature activity latency curing agent 3 - 60 weight sections, and the (C) aspect ratio use 3 or more and 500 or less whisker 0.05 - 30 weight sections as an indispensable component to following (A), (B), and the (C) (component A) epoxy resin 100 weight section.

[0012] This invention is explained more to a detail below. Especially the epoxy resin that is the (A) component used for this invention is not limited, and the bisphenol A mold epoxy resin, other glycidyl ether mold epoxy resins, for example, a bisphenol female mold epoxy resin, a bisphenol smooth S form epoxy resin and a phenol novolak mold epoxy resin, a cresol novolak mold epoxy resin, a glycidyl amine mold epoxy resin, a naphthalene mold epoxy resin, a bromination bisphenol A mold epoxy resin, a glycidyl ester mold epoxy resin, a ring type aliphatic series epoxy resin, a heterocycle type epoxy resin, etc. are mentioned. Moreover, according to a request, an urethane modified epoxy resin, a rubber modified epoxy resin, an alkyd modified epoxy resin, etc. may be used. Although the bisphenol A mold epoxy resin and a novolak mold epoxy resin are [among these] more desirable than the balance of handling nature, profitability, and composite-material physical properties, according to a need property, it can mix suitably and these two or more sorts can be used.

[0013] (B) It is the thermosetting latency curing agent which what is activated above 50 degrees C is used as a low-temperature activity latency system curing agent of a component, and is preferably activated near 80 degree C. For example, there are a latency curing agent (friend cure: trademark by Ajinomoto [Co., Inc.] Co., Inc.) of the amine adduct mold activated by thermofusion, a microcapsule type latency curing agent (Novacure: trademark by Asahi Chemical Industry [Co., Ltd.] Co., Ltd.) of a septum destructive mold, etc., these latency curing agents have good preservation stability near 50 degree C, rapid activation is carried out from near 80 degree C, and an epoxy resin is stiffened. 3 - 60 weight section is used to the epoxy resin 100 weight section whose addition of these latency curing agents is the (A) component. If property that hardening is slow and sufficient will not be acquired if fewer than 3 weight sections, but the 100 weight sections are exceeded, lowering of the preservation stability near a room temperature and lowering of hardened material physical properties are remarkable, and are not desirable. Moreover, although it is [the hardening reactivity near 80 degree C] better to use these latency curing agents independently, from the objects, such as moderate control of reactivity or preservation stability, according to a request, it can mix suitably and the urea compound of extent to which a property is not reduced, a guanidine compound, a polyvalent-carboxylic-acid poly hydrazide compound, amine imide, diaminomaleonitrile, guanamines, phenol resin, melamine resin, a urea resin, etc. can be used for them.

[0014] (C) As a whisker which is a component, 450 or less thing is preferably used for an aspect ratio (diameter of the die length/fiber of fiber) 500 or less three or more. If an aspect ratio is smaller than 3, the resin flow at the time of shaping will not become low enough, and an adverse effect will be brought to the impregnating ability to consolidation fiber or the workability of prepreg, and a moldability if an aspect ratio exceeds 500. Especially these whiskers are not limited and kinds, such as boric-acid aluminum, a calcium carbonate, carbonic acid silicon, silicon nitride, potassium titanate, basic magnesium sulfate, a zinc oxide, graphite, a magnesia, a calcium sulfate, boric-acid magnesium, 2 titanium borides, alpha-alumina, and a chrysotile, or two sorts or more are used suitably. Boric-acid aluminum, a calcium carbonate, and a silicon carbide whisker are [especially among these] excellent in respect of a property and profitability. the addition of these whiskers -- the (A) component 100 weight section -- receiving -- 0.05 - 30 weight section -- 0.5 - 15 weight section is used preferably. When fewer than the 0.05 weight section, there is no lowering of sufficient flow at the time of shaping, if 30 weight sections are exceeded, the viscosity of resin will rise, and the impregnating ability to the fiber in a prepreg chemically-modified degree is easy to fall and is not desirable.

[0015] The thermoplastics of extent to which lowering of extent to which impregnating ability to consolidation fiber is not reduced by request in addition to these above-mentioned component or reactivity, thermal resistance, storage stability, etc. is not carried out, and an epoxide reactivity diluent may be added. As an example of thermoplastics, acetal resin, such as phenoxy resin, a polyvinyl butyral,

a polyvinyl formal, and a polyvinyl phenyl acetal, polyether sulphone, polysulfone, polyether imide, polyarylate, etc. are mentioned, and phenyl glycidyl ether, butyl glycidyl ether, alkyl glycidyl ether, styrene oxide, octylene oxide, such mixture, etc. are mentioned as an example of a reactant diluent. In addition, additives, such as flame retarders, such as release agents, such as coupling agents, such as a silane and a titanate compound, a higher fatty acid, and a wax, a halogen, and phosphorus compounds, a defoaming agent, a coloring agent, and a low-temperature foaming agent, can also be used if needed. [0016] Moreover, as consolidation fiber used for manufacture of these prepregs, a carbon fiber, a glass fiber, an aramid fiber, polyester fiber, silicon carbide fiber, a boron fiber, an alumina fiber, a polyethylene fiber, etc. are mentioned, and these kinds or two sorts or more are used suitably. And especially a carbon fiber is desirable in respect of the reinforcement of the Plastic solid after hardening obtained among these, or conductivity.

[0017] Although what is necessary is to be able to apply the general prepreg manufacture approach, and to sink in by the direct or film method by the hot melt method as opposed to a reinforcement base material, or just to use either of the impregnation after film[direct or]-izing by the solvent impregnation method, in order to manufacture prepreg, a solvent distilling-off process is required of a solvent impregnation method, and the stability of a resin constituent may be reduced and it is not so desirable.

[0018]

[Example] This invention is not limited by these examples although an example explains this invention in more detail below. In addition, the code and the examining method of the compound used into the example are as follows.

<Raw material> "E828": Bisphenol A mold epoxy resin (oil-ized shell company make)

"E1001": Bisphenol A mold epoxy resin (oil-ized shell company make)

"PN-23", "MY-24": Amine adduct mold latency curing agent (Ajinomoto Co., Inc. make)

"HX-3722": Microcapsule type latency curing agent (Asahi Chemical Co., Ltd. make) [an E828/curing agent =2/3 weight section masterbatch]

DICY: Dicyandiamide (oil-ized shell company make)

DCMU: 3-(3, 4-dichlorophenyl)-1 and 1-N dimethyl urea (the Hodogaya chemistry company make)

"Alvo REXX YS2" "YS3": boric-acid aluminum whisker (Shikoku Chemicals [Corp.] Corp. make)

<aspect ratio =10-60> "WISUKARU": calcium-carbonate whisker (Shikoku Chemicals [Corp.] Corp. make) <aspect ratio =20-60> "R202": pulverizing silica and Aerosil R202 (product made from Japanese Aerosil)

[0019] <Measurement of resin flow> prepreg is cut to 100x100mm, and they are glass fabrics after a laminating and by hot press to a hole opening film and an outermost layer of drum about a laminating and the upper and lower sides in 4 ply (ply) 80 degrees C and 3.5kg/cm² It pressurizes and a resin flow is measured.

[0020] <Assessment of winding and furnace omission> prepreg was judged so that it might be set to diagonal-crossing layer (**45 degrees) 3ply and straight layer (0 degree) 3ply. The judged prepreg was wound around the mandrel which applied the release agent on the rolling table. Subsequently, the heat shrink tape was twisted with tape RABBINGU equipment. It hung by making a major-diameter side a heating furnace upwards, and hardened for 80 degrees C / 2 hours. Furnace omission is measured after cooling to a room temperature.

[0021] < handling workability: -- tuck nature and drape property > (the lamination condition [of the diagonal-crossing layer in 23 degrees C], propriety [of correction], hardness [of a hand volume], and rolling table winding back -- bounding -- etc. -- synthetic -- decision)

O -- Fitness x -- Defect [0022] <bending test (three-point bending)> ASTM D790 -- applying correspondingly -- made in equipment: Oriental Baldwin -- UTM-5T -- using -- sample configuration: (2mm in die length of 100mm, width of face of 10mm, thickness), span length: 80mm, and crosshead speed: 2 mm/min -- measurement.

[0023] <ILSS> ASTM According to D2344, it measures with equipment same as the above by sample configuration: (2mm in die length of 12mm, width of face of 10mm, thickness), span length: 8mm, and crosshead speed: 2 mm/min.

[0024] (Example 1) "E828" was carried out in 70 weight sections, the churning dissolution of the 30 weight sections was carried out for E1001 for 30 minutes at 150 degrees C, it cooled to the room temperature, and base resin was obtained. This base resin was warmed at 55 degrees C, with the agitator, "PN-23" was carried out at 20 weight sections, churning mixing of the 5 weight sections was carried out for Alvo REXX YS2 for 30 minutes at homogeneity, and the resin constituent of this invention was obtained. In this way, one direction prepreg was manufactured by the hot melt method from the resin constituent and carbon fiber (the Toray Industries, Inc. make, "a trading card T300", and elastic-modulus 24 ton/mm²) which were obtained, and the prepreg of this invention was obtained. 150 g/m² and the amount of resin of the carbon fiber superintendent officer of this prepreg were 35%. 14 ply laminating of this prepreg was carried out to the one direction, and the one direction laminate of about 2mm thickness was fabricated with the autoclave by hardening of 80 degrees C / 2 hours. The physical properties (Vf=60% reduced property) of the obtained composite material are shown in a table -1.

[0025] The handling workability (a tuck, drape property) of this prepreg was good. Moreover, there was no furnace omission in ten obtained shaft mold goods. Furthermore, although the front face of these mold goods was ground using the grinder and observed about surface defects (crack etc.), the good mold goods which do not have a defect altogether were able to be obtained.

[0026] (Examples 2, 3, 4, and 5) With the presentation of a table 1, the resin constituent and prepreg of this invention were obtained by the same approach as an example 1. The handling workability of this prepreg is good, and there is no furnace omission in mold goods, and the defect was not observed on a polish front face.

[0027] (Example 1 of a comparison) Prepreg was manufactured by the same approach as an example 1 except having not used the (C) component with the presentation of a table -1. The superintendent officers of prepreg are 150 g/m². The amount of resin was 35%. This prepreg had high tuck nature, and handling nature was poor, and the resin flow was also large, and furnace omission occurred. Moreover, generating of the crack after mold-goods polish was also remarkable.

[0028] (Example 2 of a comparison) Prepreg was manufactured like the example 1 except having used "Aerosil R202" as an inorganic bulking agent. Although the workability of this prepreg was comparatively good, furnace omission occurs, and it is uneven on the surface of mold goods, and the crack occurred in 10 median books after polish.

[0029] (Example 3 of a comparison) Prepreg was manufactured by the same approach as an example 1 except having used 4 weight sections for DICY, having used 4 weight sections for the (B) component for DCMU, and having not used the (C) component with the presentation of a table 1. Handling workability is poor, and is inadequate, and the cut of it was not completed. [of hardening]

[0030] [A table 1]

表 - 1

	実 施 例					比 較 例		
	1	2	3	4	5	1	2	3
(A) 成分「E828」 「E1001」	70 30	70 30	70 30	70 30	60 40	70 30	70 30	40 60
(B) 成分「PN-23」 「MY-24」 「HX-3722」 DICY DCMU	20 - - - - -	30 - - - - -	20 - - - - -	- 35 - - -	- - 40 - - -	20 - - - - -	20 - - - - -	- - - - 4 4
(C) 成分「アルボレックスYS2」 「アルボレックスYS3」 「ウィスカル」	5 - -	5 - -	- 5 -	- - 5	- - 5	- - -	- - -	- - -
その他「アエロジルR202」	-	-	-	-	-	-	5	-
取扱作業性 (23℃)	○	○	○	○	○	×	○	×
レジンフロー (%) 炉落ち (mm) シャフト成形品欠陥 (キズ)	3.2 0 0/10	1.8 0 0/10	2.8 0 0/10	3.5 0 0/10	3.0 0 0/10	17.3 125 4/10	10.8 12 2/10	- - -
硬化条件 80℃×2時間	○	○	○	○	○	○	○	硬化せず
0° 曲げ強度 (kg/mm ²) 0° 曲げ弾性率 (kg/mm ²) ILSS (kg/mm ²)	175 13700 8.3	172 13800 9.0	178 13800 9.5	172 13200 9.5	173 13200 9.2	163 13000 8.5	170 13000 9.0	- - -

[0031]

[Effect of the Invention] The prepreg manufactured from the matrix resin containing the whisker of this invention can adjust freely moderate workability and the resin flow at the time of shaping, and there is no defect by the fabricating methods, such as oven shaping and internal pressure shaping, and since cold cure is possible, it is suitable also for large-sized shaping, and various kinds of Plastic solids which have

the outstanding composite-material property are acquired. Thus, it is very useful to improvement in the dependability of a fiber reinforced composite material, improvement in productivity, improvement in profitability, etc.

[Translation done.]

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CLAIMS

[Claim(s)]

[Claim 1] The epoxy resin constituent for preregs characterized by (B) low-temperature activity latency curing agent 3 - 60 weight sections, and the (C) aspect ratio containing 3 or more and 500 or less whisker 0.05 - 30 weight sections to following (A), (B), and the (C) (component A) epoxy resin 100 weight section.

[Claim 2] The epoxy resin constituent according to claim 1 characterized by the low-temperature activity latency curing agent of (B) being a latency curing agent of an amine adduct mold.

[Claim 3] The epoxy resin constituent according to claim 1 characterized by the low-temperature activity latency curing agent of (B) being a latency curing agent of a microcapsule type.

[Claim 4] The epoxy resin constituent according to claim 1 characterized by the whisker of (C) being boric-acid aluminum.

[Claim 5] The epoxy resin constituent according to claim 1 characterized by the whisker of (C) being a calcium carbonate.

[Claim 6] Prepreg characterized by coming to sink into consolidation fiber in an epoxy resin constituent according to claim 1 to 5.

[Translation done.]

(19) 日本国特許庁 (J P)

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(54) 【発明の名称】 エポキシ樹脂組成物及びそれを用いたプリプレグ

(57) 【要約】

【構成】 下記(A)、(B)および(C)成分

(A) エポキシ樹脂100重量部に対し

(B) 低温活性潜在性硬化剤3~60重量部及び

(C) アスペクト比が3以上、500以下のウィスカー
0.05~30重量部

を含有することを特徴とするプリプレグ用樹脂組成物。

【効果】 本発明のウィスカーを含有したマトリックス樹脂より製造されるプリプレグは、適度な作業性と成形時のレジフローを自由に調整することが可能であり、オープン成形、内圧成形等の成形法で欠陥がなく、また低温硬化が可能のため、大型成形にも適しており、優れた複合材料特性を有する各種の成形体を得られる。この様に、繊維強化複合材料の信頼性の向上、生産性の向上、経済性の向上等にきわめて有益である。

【特許請求の範囲】

【請求項1】 下記(A)、(B)および(C)成分

(A) エポキシ樹脂100重量部に対し

(B) 低温活性潜在性硬化剤3〜60重量部及び

(C) アスペクト比が3以上、500以下のウィスカー0.05〜30重量部

を含有することを特徴とするプリプレグ用エポキシ樹脂組成物。

【請求項2】 (B)の低温活性潜在性硬化剤がアミンアダクト型の潜在性硬化剤であることを特徴とする請求項1記載のエポキシ樹脂組成物。

【請求項3】 (B)の低温活性潜在性硬化剤がマイクロカプセル型の潜在性硬化剤であることを特徴とする請求項1記載のエポキシ樹脂組成物。

【請求項4】 (C)のウィスカーがホウ酸アルミニウムであることを特徴とする請求項1記載のエポキシ樹脂組成物。

【請求項5】 (C)のウィスカーが炭酸カルシウムであることを特徴とする請求項1記載のエポキシ樹脂組成物。

【請求項6】 請求項1ないし5のいずれかに記載のエポキシ樹脂組成物を強化繊維に含浸してなることを特徴とするプリプレグ。

【発明の詳細な説明】

【0001】

【産業上の利用分野】本発明はプリプレグの適度なタック性(粘着性)、ドレープ性(柔軟性)等の取扱い作業性と室温での保存安定性に優れ、80℃付近の比較的低温で成形可能であり、また成形時のレジフローが少なく、特に釣竿やゴルフシャフト等の繊維強化樹脂製の管状成形体の成形時に炉落ちがなく、これら成形体研磨表面にキズ等の欠陥がない、優れた複合材料特性を有する繊維強化複合材に適したエポキシ樹脂組成物に関する。

【0002】

【従来の技術】炭素繊維等の繊維強化複合材料は、軽量であって高強度、高弾性率を有し、樹脂との複合体、いわゆるプリプレグの形で、例えば釣竿、ゴルフクラブのシャフト、テニスラケット等のスポーツ・レジャー分野、板バネやハニカム構造材等の工業材料、さらには自動車関連、航空機材料、船舶等の構造材料、電子材料、土木建築材料等さまざまな用途に広く用いられている。

【0003】このような繊維強化複合材料用プリプレグに要求される特性としては、適度な取扱作業性、室温における保存安定性、硬化物の機械的特性に優れる事はもとより、近年船舶や車輛のような大型構造材の成形、また木型や樹脂型を用いた成形等に関して、より低温短時間の硬化要求が望まれており、成形サイクルの短縮化、エネルギーコストの低減要求が高まっている。

【0004】ところで従来この様な繊維強化複合材料用プリプレグのマトリックス樹脂としては、エポキシ樹脂

を用いたもの(特開昭62-127317号公報、特開平2-41314号公報)、またこれらのマトリックス樹脂に、補強単繊維径の10倍以下の粒径である充填剤、例えば黒鉛粉末、カーボンブラック、アルミニウム粉末、シリカパウダー等を添加し、プリプレグ成形時のマトリックス樹脂の流動性を低下したもの(特開昭59-227931号公報)等がプリプレグの製造に用いられている。

【0005】また低温硬化用プリプレグとしては、特開平5-9262号公報、特開平6-9802号公報に、100℃以下の比較的低温で硬化する潜在性硬化剤を用いたプリプレグについて提案されている。これらのマトリックス樹脂は、従来のものと比較してプリプレグの製造時、保存安定性を低下させないために、より低温で強化繊維に含浸する必要がある、そのため樹脂粘度は低めに設定する必要がある。

【0006】特に釣竿やゴルフシャフト等の管状の繊維強化複合材料は、炭素繊維やガラス繊維を一方に引き揃えたシート状のプリプレグまたは上記強化繊維の織物にマトリックス樹脂を含浸したプリプレグを離型剤を塗布したテーパ付マンドレルに長手方向に対して、所望の設計に応じた方向に巻付け、さらにその上に熱収縮性テープをテープワインダー等を用いて少しずつオーバーラップさせながら螺旋状に巻付け、次いで加熱炉に入れ、熱収縮性テープによる締め付け力を付与しながらマトリックス樹脂を硬化し、加熱炉から取出して冷却後テープを取り除き、脱芯機等によりマンドレルを引抜いて成形している。しかし従来のプリプレグでは、成形時にマンドレルの大径側から小径側に向かってプリプレグが移動する、いわゆる炉落ちが発生するという欠点を生じていた。これは、加熱によってマトリックス樹脂が低粘度化し流動性を増すため、このような状態で熱収縮性テープによる締め付け力が加わると、マンドレルがテーパを有しているため、大径側から小径側に分力が生じ、この方向にプリプレグが移動する、いわゆる炉落ちが発生する。この炉落ちが発生すると、マトリックス樹脂や強化繊維に分布のむらができ、所望の複合材料特性が得られないばかりか、成形品の欠陥の原因または小径部の反り発生原因ともなる。

【0007】一方、特開昭57-22636号公報に、マンドレルへのプリプレグ巻き付けに先立って、糸または熱硬化性樹脂を含浸した糸をマンドレルの長手方向に螺旋状に巻付け、その摩擦により炉落ちを防止する方法が提案されている。しかし、この方法では、炉落ち防止効果が十分でないばかりか、糸が成形品中に残存し特性上好ましくない。また特開昭59-159315号公報には、マンドレルにプリプレグを捲回するのに先立って、プリプレグ用マトリックス樹脂に対して、130℃におけるゲルタイムが前記樹脂の80%以下の熱硬化樹脂をマンドレルに塗布することにより、炉落ちを防止す

る方法が提案されている。しかしこの方法では、B-ステージ化によりゲルタイムを調整した下塗用の樹脂が必要であり、成形に先立ち、この下塗り樹脂の塗布工程、風乾が必要で、著しく生産性を低下する。更にこれらのプリプレグは、100℃以上の硬化温度が必要である。

【0008】またプリプレグの作業時の取扱性（タック、ドレープ性）に関しては、シャフト捲回時の生産性のみならず、成形品表面の欠陥（キズ等）の原因ともなるため適度な特性が要求される。タック性が強すぎると捲回時にエアを巻き込みやすく、低すぎるとプリプレグの張り合わせ等の作業性が低下する。またドレープ性が不十分の場合にはプリプレグ捲回時に巻終り部分にハネを生じ、特に高弾性率の炭素繊維では折れの原因ともなる。しかるに、これらの因子がシャフト成形後の研磨、塗装時にキズ等の欠陥として表面に現れ、不良品の発生、物性の低下の原因になっており、著しく歩留まりを低下させていた。

【0009】このように従来のマトリックス樹脂組成物を用いたプリプレグでは、適度な作業性と優れた複合材料特性を有し、室温での保存安定性が良好であり、100℃以下の成形で十分硬化して、成形時の炉落ちがなく、成形品の研磨、塗装面に欠陥のないプリプレグを提供する事は極めて困難であった。

【0010】

【発明が解決しようとする課題】本発明の目的は、従来の方法の上記欠点を解決し、前処理工程を行うことなく成形時の炉落ちを防止することができ、複合材料特性を低下することなく、100℃以下の比較的低温で成形が可能であり、所望の特性を有する繊維強化複合材料成形品を得ることができる。また、本発明のエポキシ樹脂組成物を用いたプリプレグは通常の捲回温度に於て、適度な捲回作業性を有し、また成形時のレジンフローを自由にコントロールできるため、研磨後の成形品表面に欠陥（キズ等）のない優れた成形体を提供するものである。

【0011】

【課題を解決するための手段】すなわち本発明は、下記（A）、（B）および（C）成分

（A）エポキシ樹脂100重量部に対し

（B）低温活性潜在性硬化剤3～60重量部及び

（C）アスペクト比が3以上、500以下のウィスカー 40 0.05～30重量部

を必須成分とするエポキシ樹脂組成物とこれを用いたプリプレグに存する。

【0012】以下本発明をより詳細に説明する。本発明に用いられる（A）成分であるエポキシ樹脂は特に限定されるものではなく、ビスフェノールA型エポキシ樹脂、他のグリシジルエーテル型エポキシ樹脂、例えば、ビスフェノールF型エポキシ樹脂、ビスフェノールS型エポキシ樹脂、またフェノールノボラック型エポキシ樹脂、クレゾールノボラック型エポキシ樹脂、グリシジル 50

アミン型エポキシ樹脂、ナフタレン型エポキシ樹脂、臭素化ビスフェノールA型エポキシ樹脂、グリシジルエーテル型エポキシ樹脂、環式脂肪族エポキシ樹脂、複素環式エポキシ樹脂等が挙げられる。また所望に応じて、ウレタン変性エポキシ樹脂、ゴム変性エポキシ樹脂、アルキド変性エポキシ樹脂等を用いても良い。これらのうち取扱性、経済性、複合材料物性のバランスよりビスフェノールA型エポキシ樹脂、およびノボラック型エポキシ樹脂が好ましいが、必要特性に応じてこれらの2種以上を適宜混合して用いることができる。

【0013】（B）成分の低温活性潜在性系硬化剤としては、50℃以上で活性化するのが用いられ、好ましくは80℃付近で活性化する熱硬化性潜在性硬化剤である。例えば熱溶融により活性化するアミンアダクト型の潜在性硬化剤（アミキュア：味の素（株）社製商標）、隔壁破壊型のマイクロカプセル型潜在性硬化剤（ノバキュア：旭化成工業（株）社製商標）等があり、これらの潜在性硬化剤は、50℃付近では保存安定性が良好であり、80℃付近より急速活性化しエポキシ樹脂を硬化させる。これらの潜在性硬化剤の添加量は、（A）成分であるエポキシ樹脂100重量部に対し3～60重量部が用いられる。3重量部より少ないと硬化が遅く十分な特性が得られず、100重量部を超えると、室温付近での保存安定性の低下や硬化物物性の低下が顕著であり好ましくない。またこれらの潜在性硬化剤は、単独で用いる方が80℃付近での硬化反応性は良好であるが、反応性または保存安定性の適度なコントロール等の目的より、特性を低下させない程度の尿素化合物、グアニジン化合物、多価カルボン酸ポリヒドrazid化合物、アミンイミド、ジアミノマレオニトリル、グアナミン類、フェノール樹脂、メラミン樹脂、ユリア樹脂等を所望に応じて適宜混合して用いることができる。

【0014】（C）成分であるウィスカーとしては、アスペクト比（繊維の長さ／繊維の直径）が3以上、500以下、好ましくは450以下のものが用いられる。アスペクト比が3より小さいと、成形時のレジンフローが十分に低くならず、またアスペクト比が500を超えると強化繊維への含浸性またはプリプレグの作業性、成形性に悪影響をもたらす。これらのウィスカーは特に限定されるものではなく、例えばホウ酸アルミニウム、炭酸カルシウム、炭酸ケイ素、窒化ケイ素、チタン酸カリウム、塩基性硫酸マグネシウム、酸化亜鉛、グラファイト、マグネシア、硫酸カルシウム、ホウ酸マグネシウム、二ホウ化チタン、 α -アルミナ、クリソタイト等の一種または二種以上が適宜用いられる。これらのうち特にホウ酸アルミニウム、炭酸カルシウム、炭化ケイ素ウィスカーが特性、経済性の点で優れる。これらウィスカーの添加量は、（A）成分100重量部に対し、0.05～30重量部、好ましくは0.5～15重量部が用いられる。0.05重量部より少ないと成形時の十分なフ

ローの低下はなく、30重量部を超えると樹脂の粘度が上昇し、アリアレグ化工程での繊維への含浸性が低下しやすく好ましくない。

【0015】これら上記成分以外に、所望によって強化繊維への含浸性を低下させない程度または反応性、耐熱性、貯蔵安定性等の低下をさせない程度の熱可塑性樹脂、エポキシド反応性希釈剤を添加しても良い。熱可塑性樹脂の例としては、フェノキシ樹脂、ポリビニルブチラール、ポリビニルホルマール、ポリビニルフェニルアセタール等のアセタール樹脂、ポリエーテルスルホン、ポリスルホン、ポリエーテルイミド、ポリアリレート等が挙げられ、また反応性希釈剤の例としては、フェニルグリシジルエーテル、ブチルグリシジルエーテル、アルキルグリシジルエーテル、スチレンオキサイド、オクチレンオキサイド及びこれらの混合物等が挙げられる。この他、シラン、チタネート化合物等のカップリング剤、高級脂肪酸およびワックス等の離型剤、ハロゲン、リン化合物等の難燃性付与剤、消泡剤、着色剤、低温発泡剤等の添加剤も必要に応じて用いることができる。

【0016】またこれらアリアレグの製造に用いられる強化繊維としては、炭素繊維、ガラス繊維、アラミド繊維、ポリエステル繊維、炭化ケイ素繊維、ボロン繊維、アルミナ繊維、ポリエチレン繊維等が挙げられ、これらの一種もしくは二種以上を適宜使用する。そしてこのうち得られる硬化後の成形体の強度や導電率の点で炭素繊維が特に好ましい。

【0017】アリアレグを製造するには、一般のアリアレグ製造方法が適用でき、補強基材に対し例えばホットメルト法により直接あるいはフィルム法で含浸するか、溶媒含浸法により直接あるいはフィルム化後含浸のいずれかを用いれば良いが、溶媒含浸法では溶剤除去工程が必要であり、また樹脂組成物の安定性を低下させることがあり、あまり好ましくない。

【0018】

【実施例】以下に本発明を実施例によりさらに詳しく説明するが、本発明はこれらの実施例によって限定されるものではない。尚、実施例中に用いた化合物の略号、および試験法は以下の通りである。

<原料>

「E828」：ビスフェノールA型エポキシ樹脂（油化シェル社製）

「E1001」：ビスフェノールA型エポキシ樹脂（油化シェル社製）

「PN-23」、「MY-24」：アミンアダクト型潜在性硬化剤（味の素社製）

「HX-3722」：マイクロカプセル型潜在性硬化剤（旭化成社製）〔E828/硬化剤=2/3重量部マスターバッチ〕

DICY：ジシアンジアミド（油化シェル社製）

DCMU：3-（3,4-ジクロロフェニル）-1,1

-Nジメチル尿素（保土ヶ谷化学社製）

「アルボレックスYS2」、「YS3」：ホウ酸アルミニウムウィスカー（四国化成工業（株）社製）〈アスペクト比=10~60〉

「ウィスカル」：炭酸カルシウムウィスカー（四国化成工業（株）社製）〈アスペクト比=20~60〉

「R202」：微粉シリカ、アエロジルR202（日本アエロジル社製）

【0019】<レジンフローの測定>アリアレグを100×100mmに切断し、4プライ（ply）を積層、上下を穴空きフィルム、最外層にガラスクロスを積層後、加熱プレスで80℃、3.5kg/cm²加圧し、レジンフローを測定。

【0020】<捲回および炉落ちの評価>アリアレグを斜交層（±45°）3ply、ストレート層（0°）3plyとなる様に裁断した。裁断したアリアレグを離型剤を塗布したマンドレルにローリングテーブルにより捲回した。次いで熱収縮テープをテープラッピング装置により巻付けた。加熱炉に大径側を上にして吊下げ、80℃/2時間硬化した。室温まで冷却後、炉落ちを測定。

【0021】<取扱作業性：タック性、ドレープ性>（23℃における斜交層の貼合せ状態、修正の可否、手巻きの硬さ、ローリングテーブル捲回後のハネ等より総合的に判断）

○…良好、×…不良

【0022】<曲げ試験（3点曲げ）>ASTM D790に準じて、装置：東洋ボールドウィン社製UTM-5Tを用い、サンプル形状：（長さ100mm、幅10mm、厚さ2mm）、スパン長：80mm、クロスヘッドスピード：2mm/minで測定。

【0023】<ILSS>ASTM D2344に準じて、同上装置により、サンプル形状：（長さ12mm、幅10mm、厚さ2mm）、スパン長：8mm、クロスヘッドスピード：2mm/minで測定。

【0024】（実施例1）「E828」を70重量部、E1001を30重量部を150℃で30分間攪拌溶解し、室温まで冷却してベースレジンを得た。このベースレジンに55℃に加熱し、攪拌機で「PN-23」を20重量部、アルボレックスYS2を5重量部を30分間均一に攪拌混合して、本発明の樹脂組成物を得た。こうして得られた樹脂組成物と炭素繊維（東レ社製、「トレカT300」、弾性率24ton/mm²）とから一方向アリアレグをホットメルト法で製造し、本発明のアリアレグを得た。このアリアレグの炭素繊維目付けは150g/m²、樹脂量は35%であった。このアリアレグを一方向に14プライ積層し、オートクレーブで80℃/2時間の硬化により約2mm厚の一方向積層板を成形した。得られた複合材料の物性（Vf=60%換算値）を表-1に示す。

【0025】このアリアレグは、取扱作業性（タック、

ドレープ性)は良好であった。また得られたシャフト成形品10本に炉落ちはまったくなかった。更に、この成形品の表面を研磨機を用いて研磨し、表面の欠陥(キズ等)について観察したが、すべて欠陥のない良好な成形品を得られた。

【0026】(実施例2、3、4、5)表1の組成により、実施例1と同様の方法で本発明の樹脂組成物およびアリアレグを得た。このアリアレグの取扱作業性は良好であり、また成形品に炉落ちはなく、研磨表面に欠陥は観察されなかった。

【0027】(比較例1)表-1の組成により、(C)成分を用いなかった以外実施例1と同様の方法によりアリアレグを製造した。アリアレグの目付けは150g/m²で、樹脂量は35%であった。このアリアレグはタック性が高く、取扱い性は不良であり、またレジソフロ

ーも大きく、炉落ちが発生した。また成形品研磨後のキズの発生も顕著であった。

【0028】(比較例2)無機充填剤として「エアロジルR202」を用いた以外実施例1と同様にアリアレグを製造した。このアリアレグの作業性は比較的良好であったが、炉落ちが発生し、成形品の表面にむらがあり、また研磨後10本中数本にキズが発生した。

【0029】(比較例3)表1の組成により、(B)成分にDICYを4重量部、DCMUを4重量部を用い、(C)成分を用いなかった以外実施例1と同様の方法によりアリアレグを製造した。取扱い作業性は不良であり、また硬化が不十分であり切削が出来なかった。

【0030】

【表1】

表 - 1

	実 施 例					比 較 例		
	1	2	3	4	5	1	2	3
(A) 成分「E828」 「E1001」	70 30	70 30	70 30	70 30	80 40	70 30	70 30	40 60
(B) 成分「PN-23」 「MY-24」 「HX-3722」 DICY DCMU	20 — — — —	30 — — — —	20 — — — —	— 35 — — —	— — 40 — —	20 — — — —	20 — — — —	— — — — —
(C) 成分「アルボレックスYS2」 「アルボレックスYS3」 「ウィスカル」	5 — —	5 — —	— 5 —	— — 5	— — 5	— — —	— — —	— — —
その他「アエロジルR202」	—	—	—	—	—	—	5	—
取扱い作業性 (23℃)	○	○	○	○	○	×	○	×
レジンフロー (%) 炉落ち (mm) シャフト成形品欠陥 (キズ)	3.2 0 0/10	1.8 0 0/10	2.8 0 0/10	3.5 0 0/10	3.0 0 0/10	17.3 125 4/10	10.8 12 2/10	— — —
硬化条件 80℃×2時間	○	○	○	○	○	○	○	硬化せず
0° 曲げ強度 (kg/mm ²) 0° 曲げ弾性率 (kg/mm ²) ILSS (kg/mm ²)	175 13700 9.3	172 13800 9.0	178 13800 9.5	172 13200 9.5	173 13200 9.2	163 13000 8.5	170 13000 9.0	— — —

【0031】

【発明の効果】本発明のウィスカーを含有したマトリックス樹脂より製造されるアリアレグは、適度な作業性と成形時のレジンフローを自由に調整することが可能であり、オープン成形、内圧成形等の成形法で欠陥がなく、*

*また低温硬化が可能のため、大型成形にも適しており、優れた複合材料特性を有する各種の成形体を得られる。この様に、繊維強化複合材料の信頼性の向上、生産性の向上、経済性の向上等にきわめて有益である。